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BUREAU OF MINES
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INFORMATION CIRCULAR

LOW-TEMPERATURE COKE BY THE KRUPP-LURGI PROCESS

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BY

L. D. SCHMIDT

FOREWORD

This report is one of a series written by members of the Solid Fuels Mission to Germany and describes wartime developments in mining, preparation, and utilization of coal. This mission was organized early in 1945 under the auspices of the Technical Industrial Intelligence Committee, which cooperated with a counterpart British committee. In some instances, engineers from other allied countries cooperated in the investigation. The Solid Fuels Mission operated under the direction of a steering committee headed by Dr. C. J. Potter, Deputy Solid Fuels Administrator. The personnel of the mission consisted of H. F. Yancey, in charge, J. W. Buch, Thomas Fraser, L. L. Newman, and L. D. Schmidt, of the Bureau of Mines; H. H. Lowry, Director of the Coal Research Laboratory, Carnegie Institute of Technology; H. J. Rose, Vice-President and Director of Research, Bituminous Coal Research, Inc.; and F. H. Reed, Chief Chemist, Illinois Geological Survey.

Over 50 reports were written by the members of the Solid Fuels Mission. These, together with microfilms of supporting data obtained in the course of the investigations, are on file at the Bureau of Mines, Washington, D. C., where they are available for public examination. The Office of the Publication Board in the Department of Commerce is prepared to furnish photolith copies, photostats, or microfilm reproductions of all declassified reports. A Bibliography of Scientific and Industrial Reports is issued weekly by the Department of Commerce, Office of the Publication Board, and is sold by the Superintendent of Documents, Government Printing Office, Washington 25, D. C., on a subscription basis. An initial payment of $10 will provide for approximately 9 months, and subscribers will be notified when additional remittance is necessary. All subscriptions will begin with volume 1, No. 1, unless ordered otherwise. The Bibliography covers all materials and subjects and gives instructions for obtaining the desired items.

The reports of the Solid Fuels Mission relate to the following general subjects: Mining methods in the Ruhr district of Germany, with special reference to mechanical cutting and loading equipment, pneumatic packing, and Diesel mine locomotives; activities of industrial research laboratories supported by the German coal and coke industry; coal preparation in western Europe; low-temperature carbonization processes; metallurgical coke from weakly coking coals; low-ash electrode carbon from coal; new developments in the production of metallurgical coke and the utilization of gas and by-products; briquetting brown coal and bituminous coal without the use of binders; recent engineering developments in gas turbines and steam generators, with special reference to use of coal; German coal-fired heating and cooking stoves; and the commercial extraction of coal with solvents for production of synthetic liquid fuels or chemical products.

Numerous reports on the activities of German synthetic liquid fuels plants and the plants for the production of synthesis gas from solid fuels are available in the same office as the solid fuels reports.

It is planned to publish in this series of Bureau of Mines Information Circulars the more important reports or combination of reports prepared by the members of the Solid Fuels Mission. A similar series on synthetic liquid fuel activities is being prepared.

A. C. FIELDNER, Chief
Fuels and Explosives Branch
Bureau of Mines
Washington, D. C.

I. C. 7395,
February 1947.

INFORMATION CIRCULAR

UNITED STATES DEPARTMENT OF THE INTERIOR - BUREAU OF MINES

LOW-TEMPERATURE COKE BY THE KRUPP-LURGI PROCESS[1]

By L. D. Schmidt[2]

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[1] The Bureau of Mines will welcome reprinting of this paper, provided the following footnote acknowledgment is used: "Reprinted from Bureau of Mines Information Circular 7395."

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INTRODUCTION

Now that the war is over, there is a great revival in popular demand to decrease pollution of the atmosphere in our cities. Great cities, such as Pittsburgh, are starting to enforce laws against violators of smoke ordinances. The substitution of low-temperature coke for bituminous coal in household heating is a most promising field for helping to eliminate the smoke nuisance. This Information Circular is written to assist in the very worth-while effort to reduce atmospheric pollution. Herein are described recent German developments in the production of low-temperature coke. Operating and cost data are given on German commercial-scale low-temperature carbonization plants.

ACKNOWLEDGMENTS

The author is particularly indebted to Dr. H. H. Lowry, director, Coal Research Laboratory, Carnegie Institute of Technology, for his expert assistance in the assembling of data for this report. Much credit is also due to the following, who made available many of the data contained in this report: Dr. H. J. Rose, vice president of Bituminous Coal Research, Inc.; Dr. A. R. Powell, associate director of research, The Koppers Company; Dr. A. C. Fieldner and Dr. H. F. Yancey, of the U. S. Bureau of Mines; and Thomas A. Hendricks, of the U. S. Geological Survey.

GERMAN INDUSTRIAL PLANTS FOR MAKING LOW-TEMPERATURE COKE

In Germany, two commercial-size plants of Krupp-Lurgi design have been in operation for nearly 10 years. Neither is operating at present because of the general economic collapse of Germany. New data on design and operation of low-temperature carbonization plants have become available since the war.

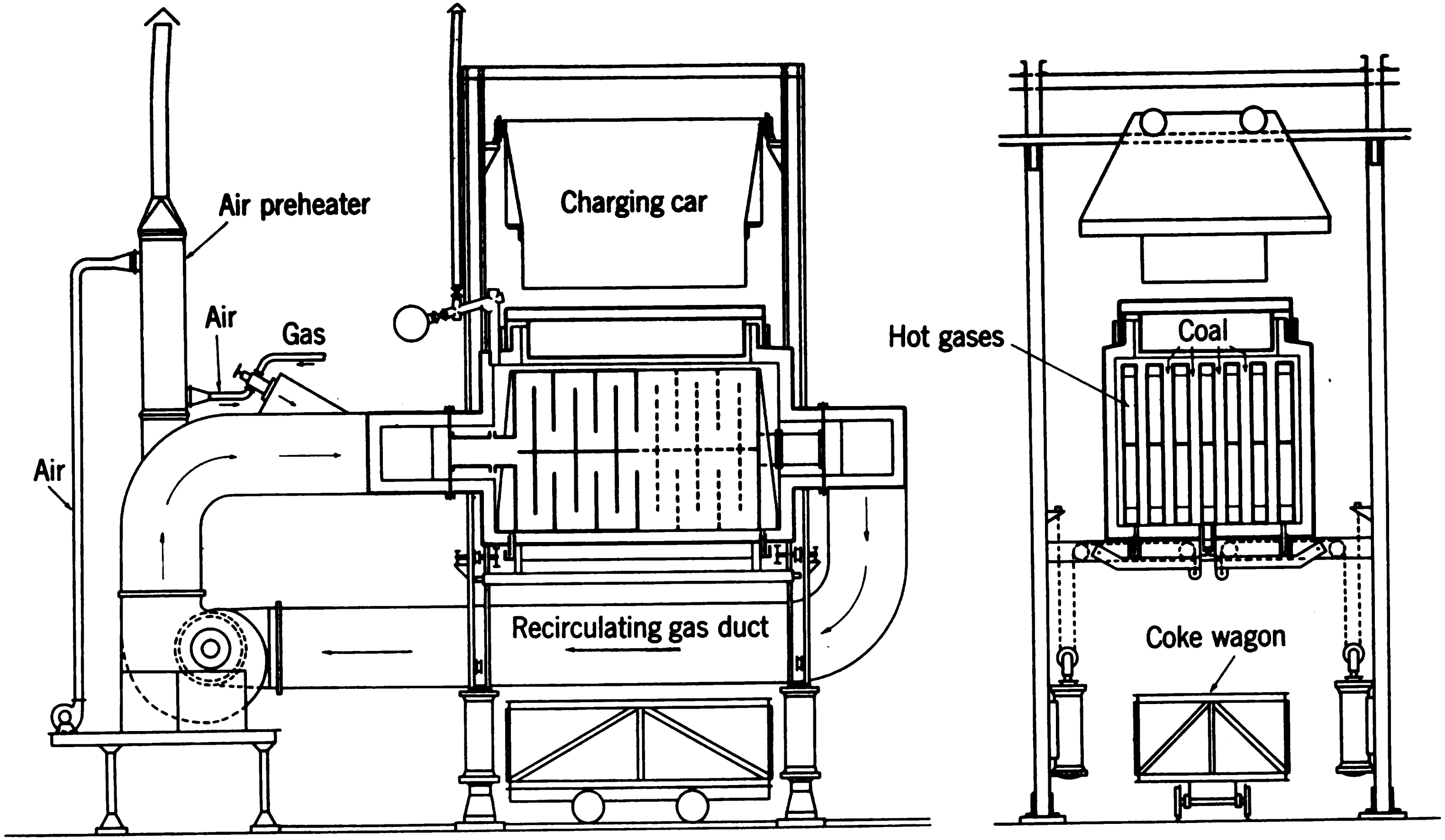

Figure 1.- Diagrammatic drawing of a Krupp-Lurgi low-temperature carbonization oven.

In the spring of 1936, two plants of Krupp-Lurgi design, each having a capacity of about 40 tons[3] of coal per day, were built - one at the Amalie mine, near Essen in the Ruhr, and the other at the Velsen mine, in the Saar. In 1937, another plant of Krupp-Lurgi design with a daily throughput of about 400 tons of coal, was constructed at Wanne-Eickel, in the Ruhr. In 1943, the plant at Wanne-Eickel was enlarged by 24 ovens with a daily throughput of 290 tons per day, making the total at the Wanne-Eickel plant nearly 700 tons of coal per day. At Velsen, in the Saar, the original plant, with a throughput of 35 tons of coal per day, was increased in 1942 to a capacity of about 140 tons of coal per day. At the end of the war, new construction at this plant was about 60 percent complete. If this new construction had been completed, the daily capacity of the Saar plant would now be 280 tons of coal per day. This rapid growth of these low-temperature carbonization plants from the original 4-ton-per-day pilot plant at the Helene mine, near Essen, proves that these ovens were successful in operation.

Other than the plants of Krupp-Lurgi design, no other really large-scale low-temperature carbonization plants for bituminous coking coal were built in greater Germany. However, it is reported that a pilot-scale plant using the Brennstoff-Technik design was put up at the Mizluwitz Colliery in Polish Silesia.[4] A large-scale, Brennstoff-Technik, low-temperature carbonization plant was started at Marienau, in the Saar, and at the time of capture by the American forces construction was about 40 percent complete.

Description of Krupp-Lurgi Low-Temperature Carbonization Plant

Figure 1 shows one of the original plants. The coal is carbonized in six narrow carbonization cells contained between seven rectangular steel heating cells, which are heated by recirculating hot gases. These six carbonization cells constitute an oven, which is filled from an overhead charging car; each carbonization cell is 10.5 feet long, 6.9 feet high, 3 inches wide at the top and 4 inches wide at the bottom. At the close of the carbonization period, the coke slabs drop from the oven into the quenching car. The oven is closed above and below with covers sitting in water seals. Figure 1 shows a cut through a heating cell and shows the method of heating the cells. The recirculating hot gases are products of a gas burner and are recirculated by means of a fan, or blower. In passing through the heating cells, these hot gases give a part of their heat to the steel walls, which conduct it directly to the coal. The velocity of the recirculation of heating gases is regulated so that the difference in temperature of the gas entering the heating cell and the gas leaving is, at the maximum, about 50° C. After these gases have passed the blower, they are again brought to the proper temperature by adding new hot products of combustion from the gas burner.

The excess hot gases pass to the atmosphere through a cast-iron heat exchanger, in which the combustion air is preheated.

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[3] 1 ton = 2,000 lb.
[4] Coke and Smokeless Fuel Age, German Low-Temperature Carbonization: 1946, pp. 77-79.

Figure 2 shows the arrangement of a later design of the Krupp-Lurgi low-temperature carbonization plant. It will be noted that the recirculating hot gases enter at the bottom of each heating cell and leave at the top. As the recirculating gases enter at about 620° C. and leave the top at about 580° C., the difference of 40° C. is sufficient to coke the additional coal at the bottom of the carbonization cell where it is 4 inches thick, compared to the top where it is only 3 inches thick.

Coals Used for Making Low-Temperature Coke

In the Ruhr, the coal charged to the ovens was a mixture of 25 percent from the Hannibal (Krupp A. G.) mine of about 22 percent volatile-matter content (Muck method) and 75 percent from the Bismarck (Gelsenkirchener Bergwerke A. G.) mine of 33 to 34 percent volatile-matter content. The coals were washed and contained an average of about 5.4 percent ash and 8 to 10 percent water, and the volatile-matter content of the mixture was about 30 percent (dry basis). Normally, in Germany, the 0- to 0.4-inch screen size of washed, mine-run coal is utilized for the production of high-temperature coke. In the Saar area, the average screen analysis of this 0.4-inch washed slack shows 48 percent through a 1/8-inch screen and 6.5 percent through a 100-mesh screen. When crushed for charging to the high-temperature coke ovens, the percentage through a 1/8-inch screen increased to 78, and the percentage through a 100-mesh screen increased to 12. The normal charge to the Krupp-Lurgi oven shows about the same screen analysis as those given for the charges to high-temperature coke ovens. The coals going to the Krupp-Lurgi ovens preferably have a screen analysis of about 77 percent minus 1/8-inch and about 5 percent minus 100-mesh.[5] The bulk density of coal of this screen analysis when charged, containing 8 to 10 percent moisture, was about 48 pounds per cubic foot. Experimental runs with finer grinding of the coal charge resulted in coke of lower strength (drum stability) and higher breeze (coke of minus 1/2-inch size) content.

In the Saar, coal from the Velsen mine was used in the Krupp-Lurgi ovens. In this mine three levels at 400, 500 and 600 meters are now productive, and the operators report that the coal in all beds on the present operating levels is uniform in character, and that all coal produced is mixed indiscriminately in the surface plants. Table 1 shows an analysis reported on the coal from the Velsen mine. Apparently, this sample had been washed and partly dried.

TABLE 1. - Analysis of coal from Velsen mine in the Saar

Percent							But/lb.	But/lb. pure coal
H_2O	Ash	C	H_2	S	N	O_2		
1.86	2.84	81.34	5.1	0.71	0.97	7.19	14,360	15,080

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[5] Bruggemann, K., Operating Results of the Low-Temperature Coke Plant of Krupp-Lurgi Design at the Amalie Mine of F. Krupp, A. G. Essen; Technische Berichte, Technische Mitteilungen Krupp, vol. 6, 1938, pp. 50-58.

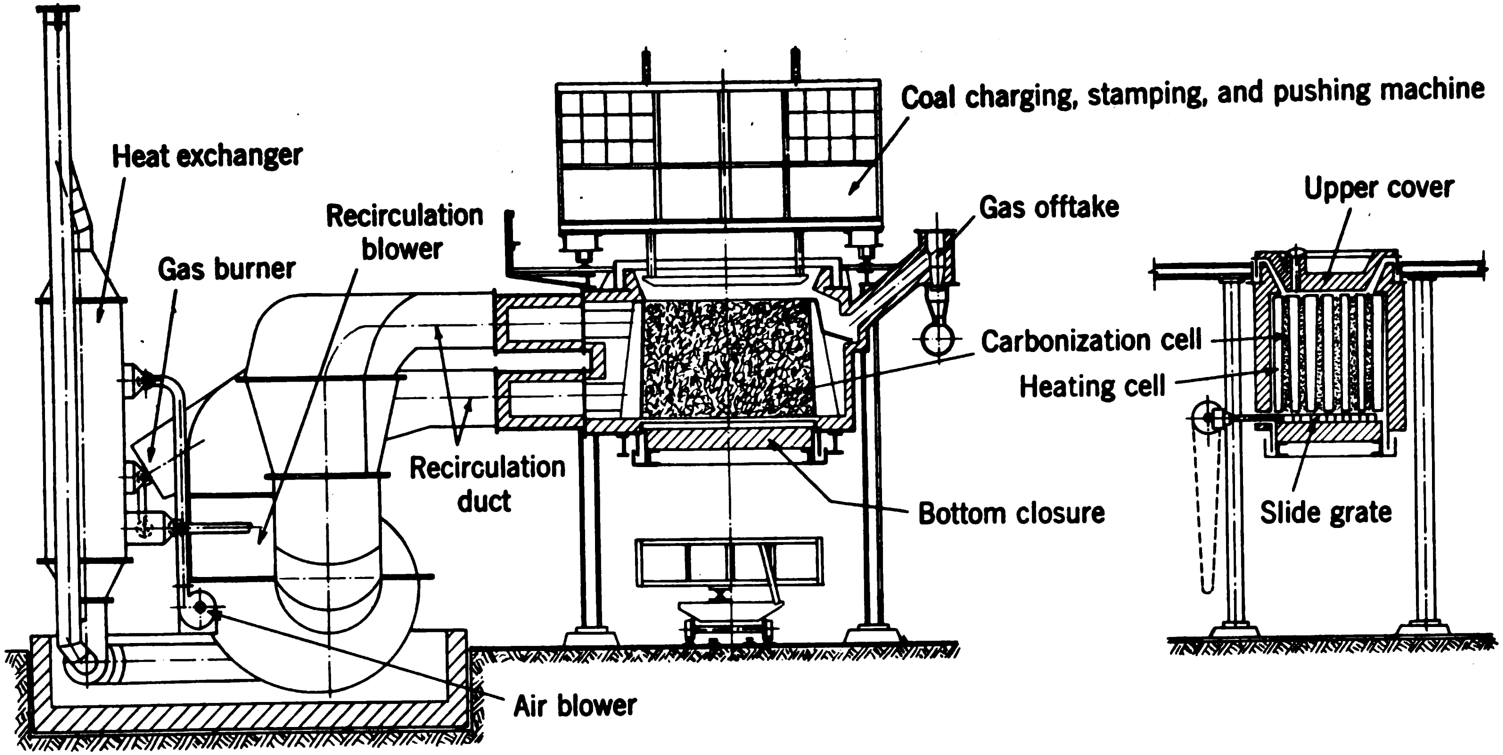

Figure 2.- Diagrammatic drawing of a Krupp-Lurgi low-temperature coke oven.

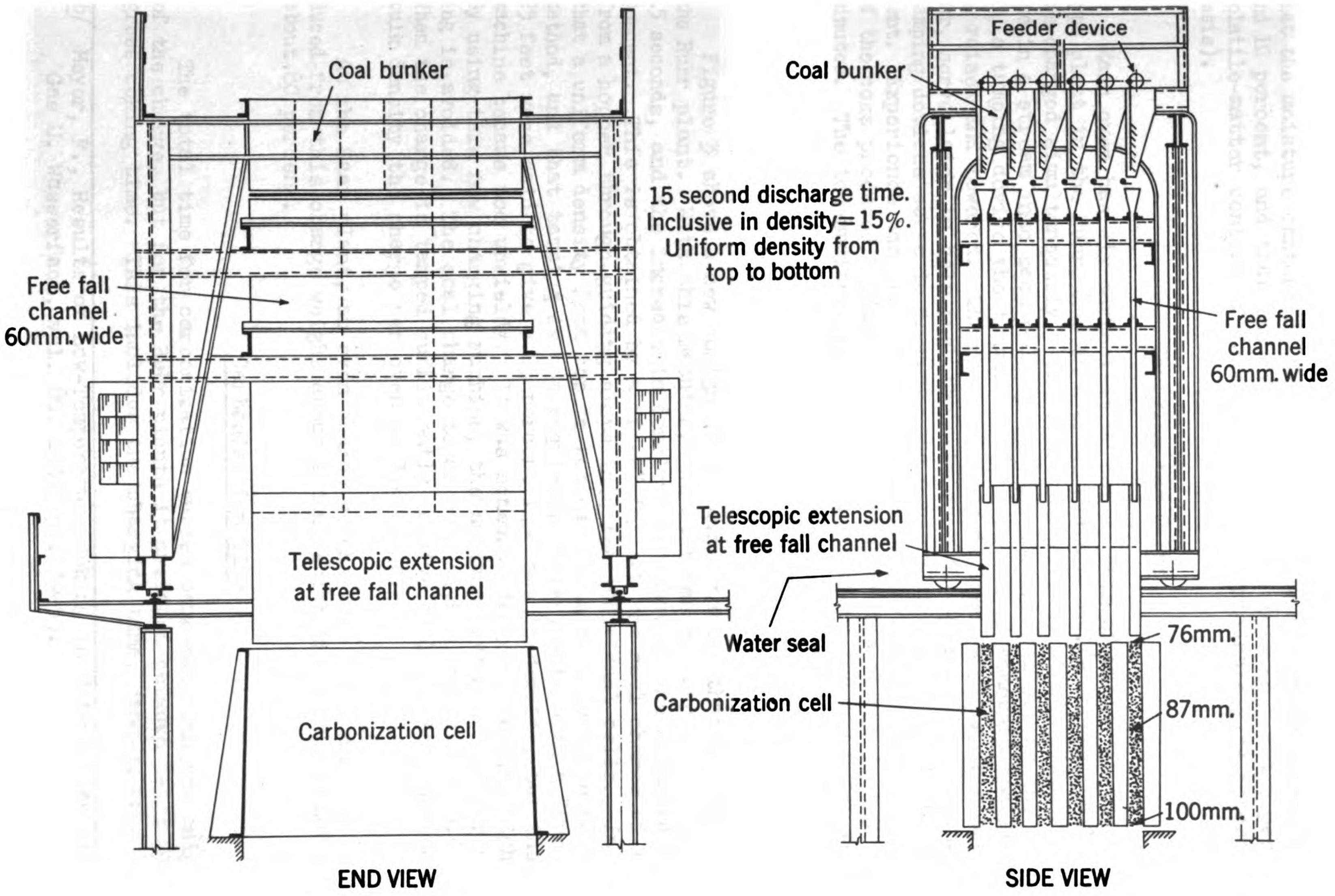

Figure 3.- Charging machine for increasing the bulk density of the coal charge by increasing the free fall of the coal.

The operators of the low-temperature coke plant at Velsen, Saar, report that the moisture content of the coal charged to the ovens ranges between 9 and 10 percent, and that the ash content ranges between 4 and 7 percent. The volatile-matter content of Velsen coal[6] is approximately 34 percent (dry basis).

Charging the Ovens

Each oven has six carbonization cells, each of which, in the latest-type plant in the Ruhr, has an average width of 3-1/2 inches. All six cells are charged simultaneously. The correctly predetermined volume of coal is fed in a stream into each cell by passing through rolls. As the coal is fed in, a tamping device the length of the oven and of somewhat smaller width is raised and lowered. These six tamping devices, working from the charging car, serve later to help push the coke from the ovens. In charging, the tamping devices serve to increase the density of the charge by about 8 percent. Experience has shown that this increased density improves the quality of the coke produced. Time required to tamp in the charge is 1.5 to 6 minutes. The top surfaces are then cleaned before the cover can be replaced on the oven; this cleaning takes about 2 minutes and is a disagreeable operation, as within about 2 minutes after the coal first enters the oven the tar fog becomes objectionable.

Figure 3 shows a new design of charging machine that has been built at the Ruhr plant. With this machine, the coal can be charged to the oven in 15 seconds, and the increase in density over that of the untamped coal is 15 percent. This is obtained by a "free" fall of the coal charge of 15 feet from a hopper through guiding slots into the individual cells. It is claimed that a uniform density from top to bottom of the cell is achieved by this method, and that tamping is not required. Experiments with a "free" fall of 25 feet were said to give a 25 percent increase in density, but the charging machine became too unwieldy. It was somewhat dangerous during high winds. By using this new charging machine, the nuisance from tar fog during charging is avoided. The coal charge to an oven without tamping is 3.0 tons. When the charge is tamped sufficiently to give an 8 percent increase in bulk density the charge per oven is 3.24 tons.

At the Saar plant, an oven charge weighs 1.26 tons, and the coke produced from this charge weighs about 1 ton, which brings the yield in coke to about 80 percent.

Carbonization Time

The total time for carbonization varies somewhat with the bulk density of the charge, but for the Ruhr plants it averaged between 5-1/3 to 6 hours gross coking time. This includes the charging time, ranging from 1.5 to 6 minutes, 2 minutes for cleaning the top surface of the ovens and closing the top cover, and 3 to 5 minutes for discharging the coke at the end of carbonization. This carbonization time corresponds to an oven-wall temperature of about 600° C.

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[6] Meyer, F., Results of Low-Temperature Coking in Modern Plants: G. W. F. Gas U. Wasserfach, vol. 80, 1937, pp. 59-55.

Oven Design

As was mentioned before, each oven contains six carbonization cells, and in the newest Ruhr plant each cell is 10.5 feet long, 6.9 feet high, 3 inches wide at the top and 4 inches wide at the bottom. The cells are separated by heating flues, the walls being ordinary boiler steel plate nearly 1 inch thick. All-welded construction is used. The heating flues are of cellular construction where the maximum dimensions of a cell is not more than about three times the thickness of the webbing or baffling. Thus, the baffles serve both as structural members and also to direct the recirculating hot gases in such a manner as to obtain uniform heating of the coal charge.

Figure 4 is a scale drawing showing the section through the top of the Krupp-Lurgi low-temperature carbonization oven. Figure 5 is a scale drawing showing details of construction of a battery of four ovens, which are heated by one stove. Figures 6 and 7 are photographs showing two views of the elliptical openings through which the recirculating hot gases enter and leave the heating cells. Due to the taper of the carbonization cells, the taper is in a reverse direction in the flues, which, of cource, are narrower at the bottom than at the top.

The steel used has a strength at room temperature of about 71,000 pounds per square inch, and this strength decreases rapidly above the 300° C. until at 600° C. it is reduced to about 1 percent, or 710 pounds per square inch.

It is interesting to note that in publications appearing in 1938, the authors discuss controlling the temperature at the entrance of the cells for temperatures not exceeding 630° to 640° C. Later information indicates that 620° C. at the entrance is the recommended operating temperature. The operators report that some difficulty was encountered with distortion of the walls of the end flues, but this was remedied by adding special support within the refractory housing that surrounds the metal chambers. Although samples of the steel were taken periodically for metallurgical inspection and test, little carburization, decarburization, or oxidation was observed. The tests showed that the metallographic structure of the steel did not change appreciably with time. Some of the ovens were not cooled for 5 or 6 years. The wall thickness decreased about 0.02 inch per year, and experience indicated a life of 10 or 12 years could be anticipated. The latest plant in the Ruhr used steel for the oven walls 0.95 inch thick. The earlier Saar plant used steel 0.89 inch thick.

To help preserve the metal walls, an oxygen recorder was used to help control the oxygen content of the recirculating hot gases to maintain the oxygen content between 0.8 and 1.5 percent.

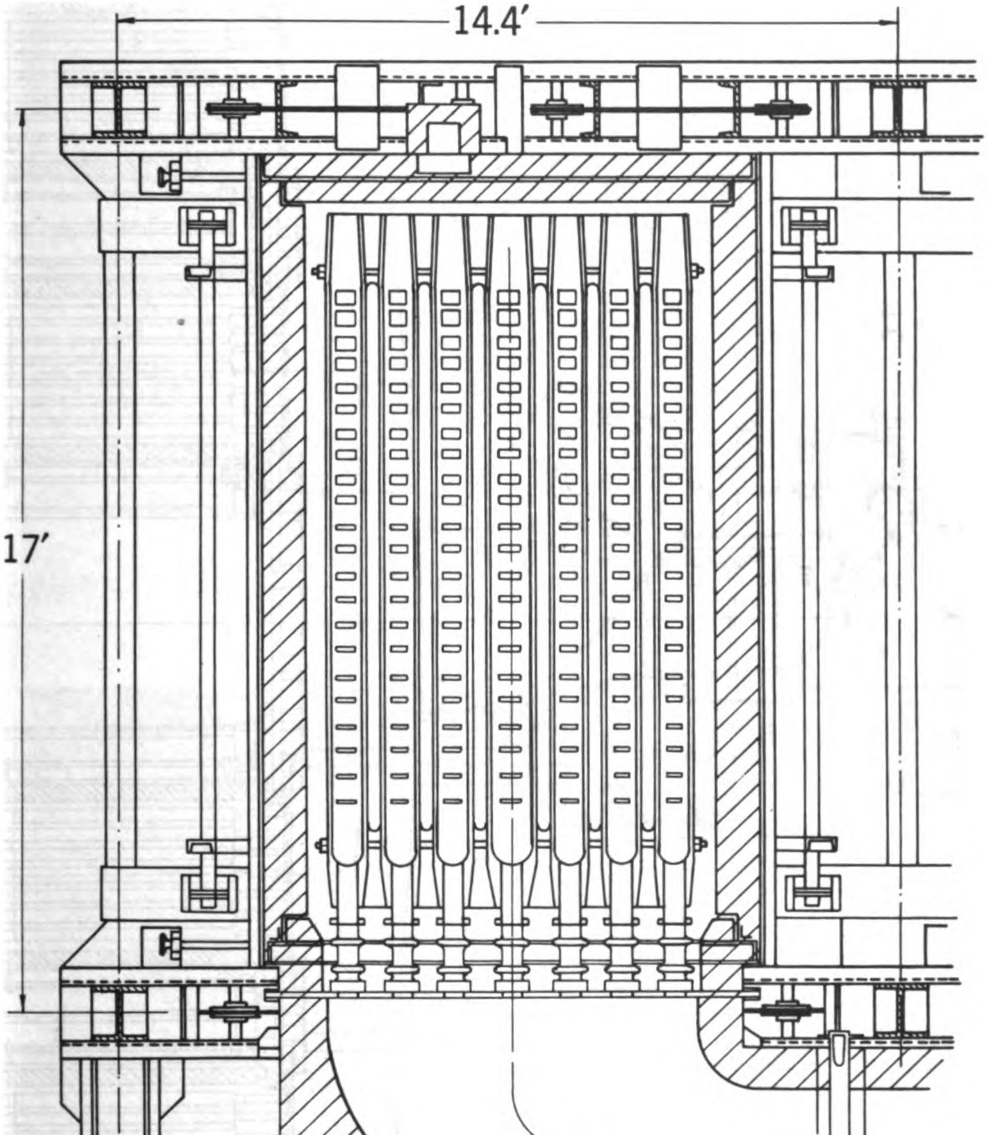

Figure 4.- Section through top of Krupp-Lurgi low-temperature carbonization oven.

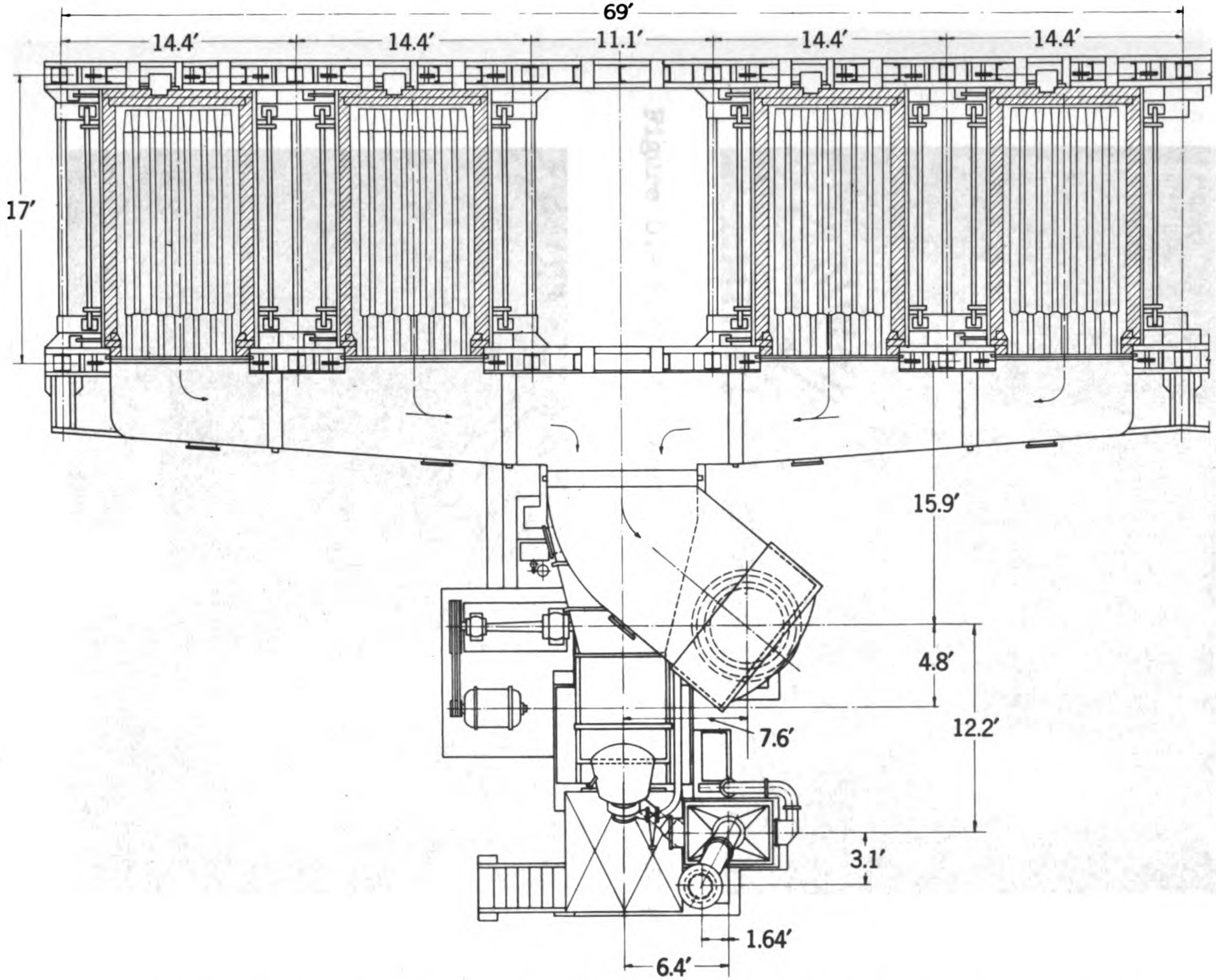

Figure 5.- Plan of Krupp-Lurgi low-temperature carbonization plant.

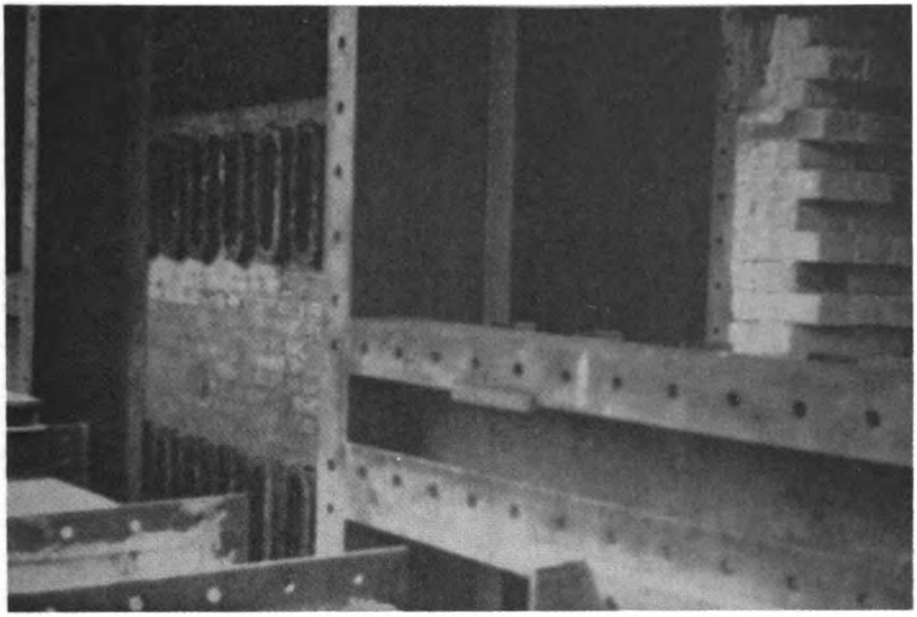

Figure 6.- Ports for entry and exit of heating gases.

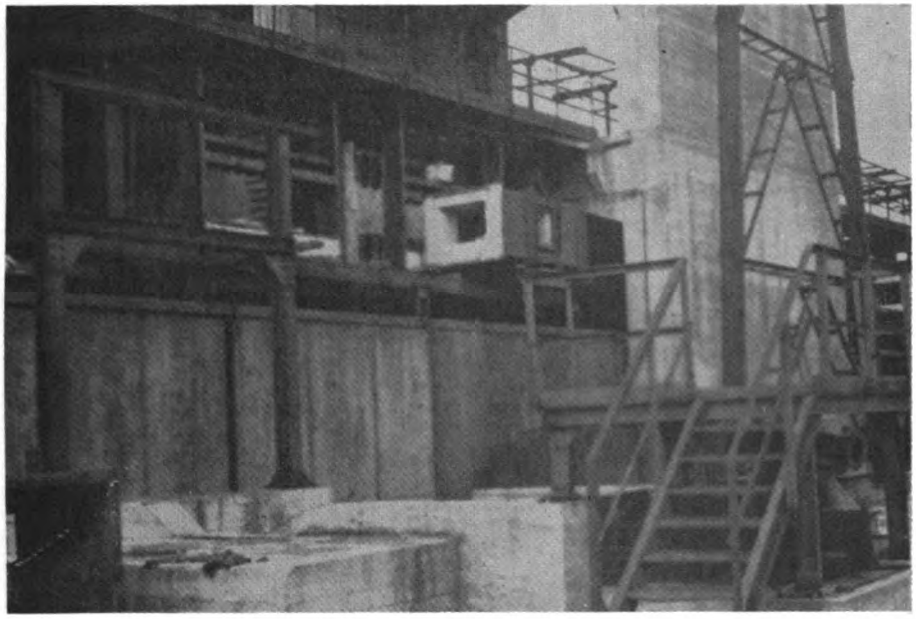

Figure 7.- Recirculating gas ducts.

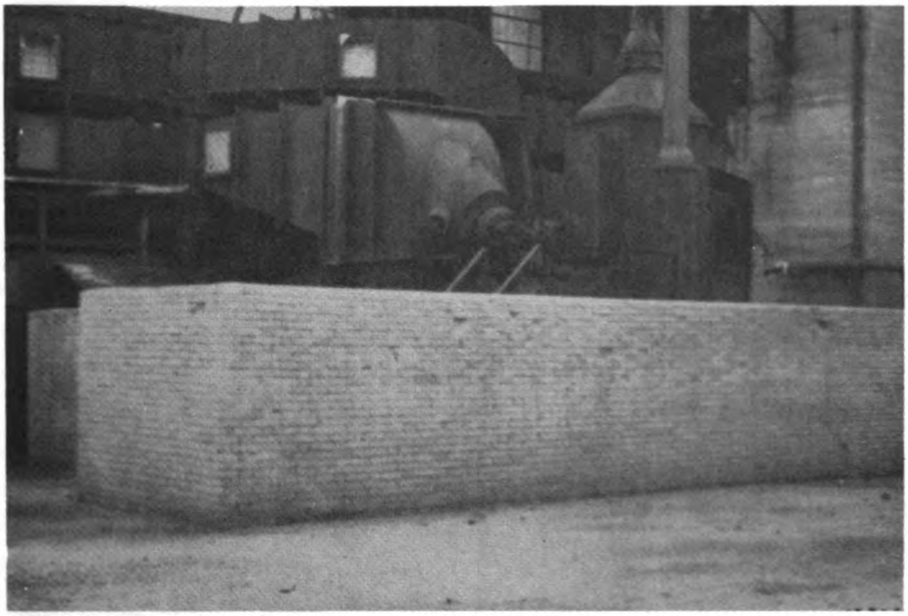

Figure 8.- Gas burner and air preheater.

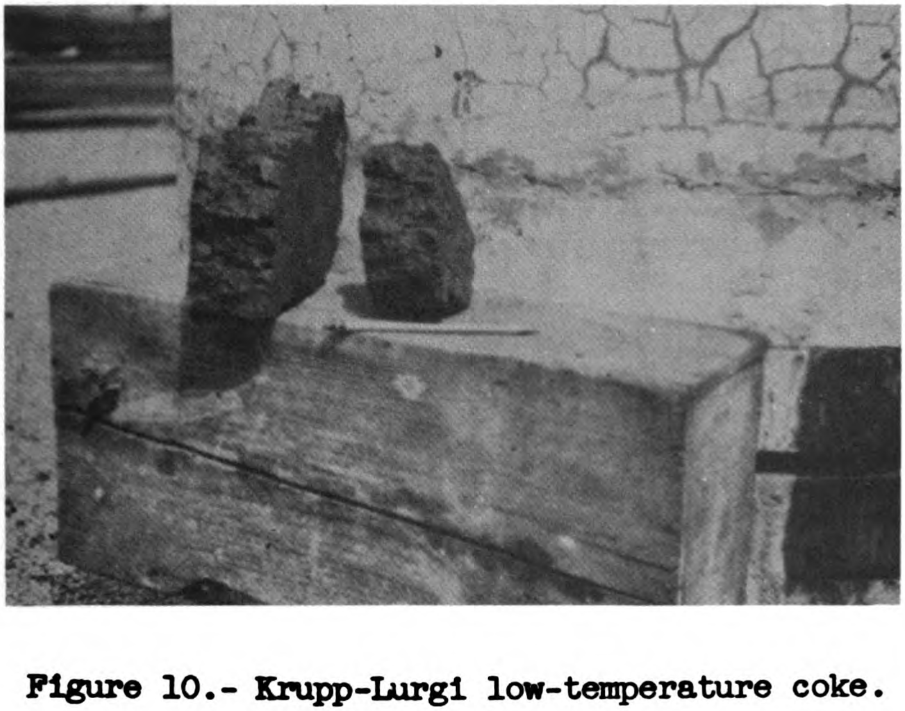

Figure 10.- Krupp-Lurgi low-temperature coke.

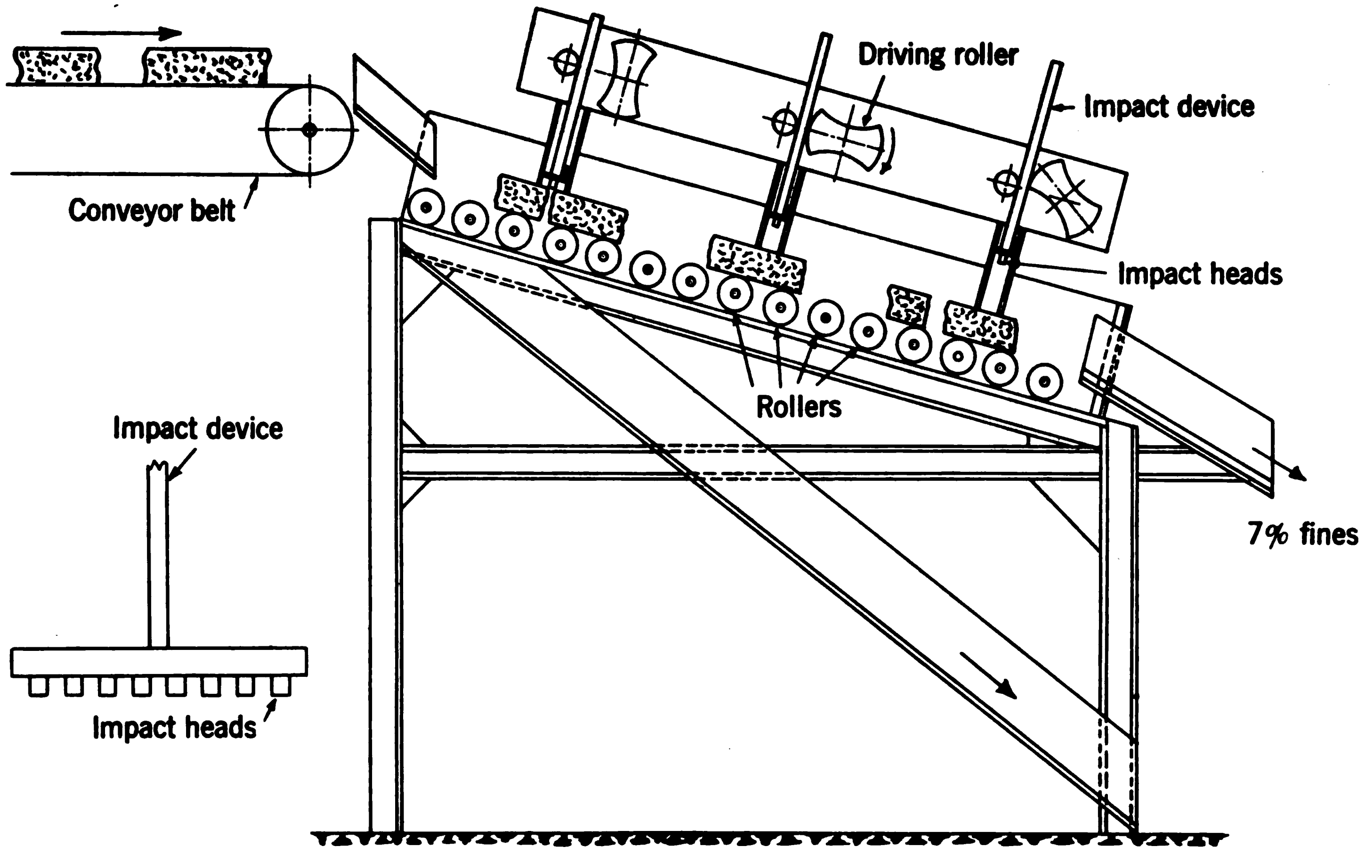

Figure 9.- Coke breaker.

Sometimes, for one reason or another, local buckling of the oven wall occurs, and the operators at the Ruhr plant have developed two types of equipment, one mechanical and the other hydraulic, to flatten the metal walls. They began to use this equipment after the ovens were 2 or 3 years old.

Figure 2 shows details of the coal-supporting slide grate that was built into the newest oven. This slide grate or grid serves to hold the coal charge in the ovens by closing the bottom of the carbonization cell so that the bottom door serves simply as a gas seal and does not support the coal in any way. Before the coke is pushed, the grate is moved far enough so that the openings in it correspond with the carbonization cell, and thus the coke is free to fall out the bottom of the oven.

At the Saar plant, the average width of the carbonization cells is 2-3/4 inches, instead of the 3-1/2 inches found at the latest Ruhr plant. The reason for the narrower ovens in the Saar is that the Velsen coal carbonized there is less strongly caking than are the Ruhr coals. More rapid heating tends to make a better coke of the more weakly caking coals.

Heating the Oven

In the newest ovens in the Ruhr, the gas used for heating the ovens was the residual gas from the Fischer-Tropsch synthetic liquid fuel plant. This residual gas had a calorific value of about 200 B.t.u. per cubic foot. The temperature of the recirculating gases where they enter the oven is maintained constant at 620° C. by automatic control on the pressure on the combustion gas going to the burner. Temperatures measured in the heating cell averaged about 595° C. Even at the maximum heat demand at the beginning of the coking period, the temperature measured in the heating cell was only 16° C. less than at the end of the coking time.

When low B.t.u. gas is used for heating the ovens, it is customary to preheat the gas also before sending it to the burner.

To insure long life of the steel oven walls, it is important to avoid excess air for combustion in the burner in order to maintain a reducing atmosphere in the recirculating hot gases. On the average the gas recirculates about 12 times, that is the makeup gas for maintaining the temperature is about one twelfth the volume of the recirculating gases.

The fan for recirculating the hot gases is built of special chrome steel and has shown no corrosion or trouble due to burning away. Figure 8 is a photograph showing the gas burner, brick-lined ducts for the recirculating gases, and the air preheater. The low brick wall surrounding the burner or stove was built to lessen probability of bomb damage. The heat consumption with these low-temperature carbonization ovens is about 900 to 1,000 B.t.u. per pound of wet coal (10 percent water) carbonized. This compared with a heat requirement of 960 B.t.u. for a similar coal carbonized in Otto high-temperature coke ovens.

Pushing the Coke

At the end of the carbonizing period, a little over 5 hours after charging, the bottom door of the oven is lowered, the grate arrangement is moved so that the slots of the grate correspond with the slots in the oven, the top cover is removed, and, if the coke does not fall by itself into the quenching car under the oven, it is started by a push from above. The coke falls into the quenching car in large slabs, most of it being in slabs several square feet in area and 3 to 4 inches thick. Normally, the operators report very little pushing difficulty. Some work has been done with dangerously expanding coals in the low-temperature carbonization oven. Of the Ruhr coals, those considered to be in the class of highly expanding coals fall into the range of 18 to 21 percent volatile matter. Experiments were made by charging one such coal of 19.5 percent volatile matter, which is well known as a strongly expanding, nonshrinking, coal. This particular coal can be used in high-temperature coke ovens only after being very finely pulverized, and can be used only when special precautions are taken to maintain low bulk density in the oven. If used otherwise, pushing is very difficult and the oven walls show damage after a short time. This coal was charged to the low-temperature carbonization ovens without further pulverization and without lowering the normal bulk density. No effect was noted on the walls of the carbonization cells, and the coke could be pushed easily.

Coke Handling

In the Saar, as a temporary expedient, the coke was quenched by hauling the quenching car through a tank of water with a cable. The operators reported that this system was not satisfactory and that it was put in only because of a scarcity of funds to cover the construction of an ordinary quenching station. As the coke pieces from the oven are too large for commercial utilization, it is necessary to crush them to usual commercial sizes. Because of the extensive crushing necessary to obtain this size reduction, ordinary crushing procedures led to the production of excessive amounts of fine coke or "breeze." To lessen breeze production, a special coke-breaking installation was designed and built. Figure 9 is a diagrammatic sketch of this coke breaker. As shown, the coke is broken by impact from rams. The operators report that with this contrivance, only 9 percent of the total coke is less than 1/2 inch in size, as compared with 22 percent when a roll crusher is used. However, from published screen analyses of commercial low-temperature coke, it appears that 12 percent of minus 1/2-inch breeze would be a good average production figure. Ordinarily, about 70 percent of the crushed coke remains on a 2-inch screen.

Coke Properties

Figure 10 is a photograph of two typical pieces of low-temperature coke made by the Krupp-Lurgi process. Normally, there is no evidence of any center line or tendency to divide at the plane midway between the oven walls. In other words, the pieces are uniform from oven wall to oven wall and do not break into "half oven-length" pieces, as does high-temperature coke.

In Germany, the universally used method of evaluating the physical properties of coke is the trommel or drum test. This test is made in a drum 1 meter long and 1 meter in diameter, which is rotated for 100 revolutions with a charge of 110 pounds of the coke being tested. The drum stability is then reported as the percentage of the coke remaining on a 1.58 inch screen. With high-temperature coke, a drum stability of 70 percent is considered adequate for blast-furnace coke.

Low-temperature coke made in the 2-3/4 inch ovens in the Saar ranged in drum stability from 50 to 80, the apparent specific gravity was 0.8, and the percent porosity 42 to 45. The large sizes of coke contained about 12 percent volatile matter, whereas the fine sizes contained 16 to 18 percent volatile matter. Typical coke made in the 3-1/2-inch ovens in the Ruhr plants has the following characteristics: Ash, 3.8 percent; volatile matter, 9.1 percent; carbon (ash-free basis), 91.5 percent; hydrogen (ash-free basis), 3.8 percent; sulfur (ash-free basis), 1.0 percent; gross heating value (ash-free basis), 14,800 B.t.u. per pound; ignition point, 396° C.; porosity, 50 percent; drum stability, 76.5 percent. Compared to high-temperature coke, there is a great difference in the volatile-matter content, reactivity, and the higher hydrogen content of the low-temperature coke, which can be as high as 53 percent of the hydrogen content of the original coal, as compared with 15 percent for high-temperature coke.

Since about 12 percent of the total production of low-temperature coke is minus 1/2-inch breeze, it is fortunate for the economies of the process that, in the Saar at least, there is a good demand for this low-temperature breeze for admixture with their coals going to the high-temperature coke ovens. High-temperature coke-oven charges of 100 percent Saar coal yield coke of a quality too low for use in blast furnaces. However, if blended with finely pulverized coke breeze or, preferably, with finely pulverized low-temperature coke breeze, a satisfactory coke can be produced. For example, the drum stability of high-temperature coke made from 100 percent Heinitz coal is 36 to 40 percent. This compares with 80 for an 80-20 mixture of Heinitz-Ruhr coal, 50 to 60 for a mixture of 94 percent Heinitz coal with 6 percent of finely ground high-temperature coke breeze, and 70 to 80 for a mixture of 84 percent Heinitz coal with 12 percent low-temperature coke breeze and 4 percent high-temperature coke breeze. Thus, experience at the high-temperature coke plants in the Saar show that by use of coke breeze in the mixture coke can be made that is nearly as good as that made by blending with special Ruhr coal.

Byproduct Recovery

In the Ruhr, the raw gas enters the primary cooler at about 190° C., and the heavy tar is collected therein. The gas then goes to a Lurgi-Cottrell precipitator at about 150° C. for removal of tar fog and to an indirect cooler for collection of light tar. The pressure drop through primary cooler, electro filter, and indirect cooler is 16 inches of water.

The three tars are combined into low-temperature tar (Heizöl "S") and, after removal of the benzine, amount to 6.4 percent of the dry coal charged. Although the yield, analysis, and physical properties of the tar depend on the coal charge, a typical analysis for low-temperature tars from Ruhr coals is as follows: Specific gravity at 20° C., 1.06; Engler viscosity at 20° C. 40°; softening temperature, 5° C.; flash point, 96° C.; insoluble in benzene 0.7 percent by weight, and in aniline, 0.15 percent by weight; tar acid content, 30 percent by volume; pitch of 70° softening point, 35 percent by weight; and gross heating value, 16,650 B.t.u. per pound. The distillation analysis of the tar is as follows:

°C.	Percent by volume
98	0
170	1.5
200	4.2
230	23.0
270	40.0
300	53.0
360	66.0

The heavy tar, when dried to less than 1 percent water content, was used as a fuel by the German Navy without further refining. The miscibility of the low-temperature tar with other fuel oil has been studied and the results published.[7]

After the removal of tar, the gas goes to the ammonia washer, and the dilute ammonia liquor is used for quenching the coke. The gas may then be either sent directly to a gas holder or compressed to 10 atmospheres, cooled, freed of sulfur, and then passed through a Feld washer for recovery of benzine. The wash oil used in the Feld washer is the light tar condensate from the indirect cooler that has been debenzinized. The benzine recovered amounts to 1.25 percent, based upon the dry coal charged, and in the Ruhr has the following properties: Specific gravity at 20° C., 0.820; octane number, 90; phenol content, 0.8 percent by volume; base content, 0.5 percent by volume; aromatic content, 50 percent by volume; olefine content, 15 percent by volume; distillation begins at 50° C., and 25 percent by volume distills up to 100° C. and 96 percent up to 195° C. The Feld washer reduced the benzine content of the gas from 70 grams per normal cubic meter to 3. The gas yield amounted to 3,200 cubic feet (60° F. and 30" Hg) per ton of dry coal, and a typical analysis of the gas produced from Ruhr coal is CO_2, 2.6 percent; saturated hydrocarbons (S.K.W.), 2.4 percent; oxygen, 0.0; carbon monoxide, 3.8 percent; hydrogen, 25.7 percent; CH_4, 59.5 percent; N_2, 6 percent; gross heating value is 830 B.t.u. per cubic foot.

With regard to the differences in yields between high-temperature and low-temperature coking, the following data are illuminating: For 100-pounds of dry coking coal with, for example, 28 percent volatile matter content, one obtains, by low-temperature coking, 6 pounds of fuel oil; with high-temperature coking (corresponding to a tar yield of 3.75 percent), 1.5 pounds (including naphthalene and anthracene).[8] Much of the high-temperature tar, all of which must be processed, comes from the distillation step as pitch, for which there is relatively little demand. Adding the light oil yields, then, from low-temperature coking, the total usable liquid fuels amount to 6.6 pounds, as compared with 2.4 pounds with high-temperature coking, when the charge is 100 pounds of dry coal. With regard to phenols and cresols, Demann reports that low-temperature distillation gives about 10 times as much as high-temperature coking....

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[7] Domann, W., Miscibility of Fuel Oils; Gluckauf, 1940, pp. 61-68.

In the Saar plant, the tar yield amounted to about 8 percent of the coal (dry basis), and this tar was obtained in three fractions. The first fraction, in the pipe line from the oven to the primary cooler, was heavy tar amounting to about 0.7 percent of the weight of the coal; the second fraction, from the primary cooler, called middle tar, amounted to about 6 percent of the weight of the coal; and the final tar, or light tar, amounted to 1.3 percent. Total water content of the tar was not more than 1.5 percent.

Cost of Plant and Operation

It is reported that the entire cost of the Krupp-Lurgi plant at Wanne-Eickel was about 2 million dollars,[9] 32 ovens were built in 1937, when labor was relatively cheap, and 24 ovens in 1943, when labor was relatively expensive. A new plant of 24 ovens and the direct equipment for this operation cost about 800 thousand dollars.

About 100 men are required to operate it: 8 men on each of three shifts for the 24 new ovens, an equal number on the 32 old ovens, 1 man on each of three shifts in the bunker house, and the rest in the byproduct plant. Wages paid were $3.20 to $4.40 per day.

At the time when the Saar plant had a capacity of about 35 tons per day, it was reported that the plant required 3 workers and 1 foreman for operation.

Table 2 gives data on the cost of operation of the Krupp-Lurgi low-temperature plant in the Saar. The relative cost and realization on the products are given for both high-temperature and low-temperature coke.

In this comparison of the cost of high-temperature and low-temperature operation, it should be remembered that the scale of the two plants is much different; that is, the high-temperature coke plant had a through-put of several thousand tons per day, whereas the low-temperature coke plant was based on a plant with a through-put of less than 150 tons per day.

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[8] Demann, W., Low-Temperature Coke as Raw Material for Chemical and Metallurgical Processes: Gluckauf, 1937, pp. 1101-06.

[9] 1 Reichsmark equals $0.40.

TABLE 2. - Comparison of cost and realization on products of high-temperature coking and low-temperature coking at Velsen in the Saar

Item	High-temperature coke	Low-temperature coke
Yield (dry basis):		
Coke, percent	72	80
Tar, percent	4.5	8.5
Gas, cu. ft. per ton	-	-
Liquor, percent	1.19	1
Ammonia, percent	0.80	-
Cost (basis; 1 ton coke, 5 percent moisture):		
Coal	$8.45	$7.35
Labor	0.86	1.62
Steam	0.31	0.22
Power	0.09	0.14
Water	0.25	0.03
Materials	0.43	0.47
Auxiliary materials	0.29	-
Amortization	0.90	3.42
Overhead	0.36	0.43
Total	11.94	13.68
Realization on products (basis, 1 ton coke, 5 percent moisture):		
Coke	$9.72	$8.94
Tar	1.01	4.50
Gas	0.83	-
Benzol	2.16	0.72
Ammonia	0.32	-
Total	14.04	14.16
General costs:		
Power, per kw.	$0.01	$0.01
Water, per ton	0.018	0.018
Steam, per ton	0.072	0.072
Coal, per ton	5.95	5.95

The low-temperature coke in the Saar in the size 3 inches by 3/4 inch sold for from $9.35 per ton to $10 per ton. This compares with the price for high-temperature coke of $8.65 per ton. The low temperature coke in the 0 to 3/4-inch sizes sold for $5.95 per ton.

Utilization of Low-Temperature Coke

In Germany, some low-temperature coke was used in locomotives and steam trains and resulted in smokeless firing. The higher reactivity of the coke made it especially suitable for suction gas producers on automobiles. It was especially useful, also, in Germany in stationary Diesel plants, where it served as a gas-generator fuel. In these plants, it is reported that 2 kilograms of low-temperature coke replace 1 kilogram of Diesel oil.

In the Ruhr, at the Wanne-Eickel plant, two of the ten water-gas generators producing synthesis gas for the Fischer-Tropsch plant for synthetic liquid fuel operated exclusively on low-temperature coke. It was claimed that this coke gave water-gas with a 1.35 ratio of hydrogen to carbon monoxide, which is considerably better than is obtained from high-temperature coke. Such gas would, of course, require less conversion by the shift catalyst to make synthesis gas. Only large coke was used in the water-gas machine and the small coke was sold for house heating for which purpose it proved to be an excellent fuel. The operators of the Wanne-Eickel plant in the Ruhr state that the combination of low-temperature carbonization with the Fischer-Tropsch synthesis plant gave a total oil yield more than 30 percent greater than the combination high-temperature coke with the Fischer-Tropsch plant. Another suggested use for low-temperature coke is in the production of ferro-silicon with high silicon content. With high-temperature coke, the silicon content is apparently limited to 77 percent but by the addition of wood charcoal, petroleum coke, or low-temperature coke the silicon content can be raised to 90 percent or more.10/ It has also been proposed that the high reactivity of low-temperature coke would make it of interest to the manufacturers of calcium carbide.

In this country, of course, the major interest in low-temperature coke would be for household heating. In the size from 3/4 inch to 1-1/2 inches, German tests indicated that the low-temperature coke made an excellent fuel for ordinary household stoves.11/

Since German practice has shown that virtually all the coals in the Ruhr, covering a wide range of coking properties, made satisfactory coke in the Krupp-Lurgi ovens. This process shows promise as a method by which cities with outstanding smoke problems can utilize the cheapest coals in their area. The high reactivity of this low-temperature coke makes it easy to ignite, quick to pick up the heating load, and it is reported to hold the fire well.

General Principles Involved in Krupp-Lurgi Process

The salient features in the Krupp-Lurgi design include (1) carbonization of the coal in a static state while confined in a narrow space between stationary, indirectly heated walls of steel; (2) use of a method of heating that is subject to precise temperature control; (3) the greatest possible avoidance of moving parts operating at carbonizing temperatures.

In general, low-temperature carbonization by means of rotating drums or ovens, in which the coal mass is agitated, or stirred, or kept in motion, has a tendency to form coke of small sizes and of low physical strength and a low-temperature tar that is so contaminated with dust that expensive processing procedures must be employed in order to use the tar.12/ When

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10/ See footnote 8.

11/ Mueller, F., The Manufacture and Use of Low-Temperature Coke: Zeitschrift des Vereines deutscher Ingenieure, 1926, pp. 1605-11.

12/ See footnote 6.

strongly coking coals are heated, they become plastic and tend to foam. The stationary walls of the Krupp-Lurgi oven exert a force or pressure that results in a more dense and stronger coke than would be obtained with free swelling in other processes. With regard to the use of steel for oven walls, the designers of the Krupp-Lurgi ovens feel that in order that the steel walls may have a practical length of life, it is essential that local overheating or direct impingement of gas flames on the steel must be avoided. Also, it is important to avoid excessive temperature changes in the oven walls during the cycle between charging and pushing. As the strength of steel at carbonizing temperatures is less than 1 percent of its value at room temperature, it is important that stress or wear on such steel parts be minimized in the design of the process.

There are some rather fundamental differences between the high-temperature and low-temperature coking processes. In high-temperature coking, rather sharply defined and narrow plastic zones formed by the melting coal move steadily inward as the time after charging increases, and the plastic zones finally meet in the middle of the oven at the end of the carbonization process. Most of the gas developed comes from the high-temperature side of this plastic zone. In the narrow Krupp-Lurgi low-temperature oven there is no well-defined plastic zone, but the whole oven content is in a plastic stage at almost the same time. The designers[13] of these ovens believe that the formation of a good low-temperature coke depends on the rapid rise of temperature in the plastic zone (350°-450° C.) of the coal. The more rapid the temperature rise in this range, the more closely all the components of the coal melt together simultaneously to form a strong coke.[14]

Table 3 contrasts the manner of temperature rise at the point midway between walls of the narrow, low-temperature coke ovens and at the midpoint in normal high-temperature coke ovens. It will be noted that the temperature in the middle of the low-temperature ovens starts to rise immediately after charging; and although the coal has 8 to 10 percent water in it, the temperature rises through the 100° C. temperature without any observable flat zone in the curve. The operators of the low-temperature ovens report that coal with 8 percent water content has been observed to be completely dry one half hour after charging. Special tests have shown that although the water is driven from coal in half an hour, nevertheless, after 1 hour almost the entire coal content of the low-temperature oven still exists unchanged as coal. Only at the walls has the coal begun to soften. However, at the end of 2 hours, almost the whole coal charge is in a plastic state. Only in the very middle can a thin sheet of unchanged coal be seen. After three hours the whole coal charge is completely plastic. This can be seen from the temperatures in table 3, where, after 3 hours, it is noted that the temperature is 430° C. at the point midway between the oven walls. In other words, the whole charge has a temperature above 430o C. and, therefore, has been converted to low-temperature coke.

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[13] This type of oven was developed by Dr. Ing. J. Goller with the encouragement and support of Dr. Ing. Fritz Mueller, director, F. Krupp, Inc.

[14] See footnote 5.

TABLE 3. - Contrasting temperature effects in high- and low-temperature coking

Time, hours	0	1	2	3	4	5	8	12	16	20	24
Temperature in middle of low-temperature oven, °C.	20	240	360	430	490	530	600	600	600	600	600
Temperature in middle of high-temperature coke oven, °C.	20	100	100	100	100	100	100	120	510	870	1000

It has been suggested that as the coking time varies with the 1.7 power of the oven width,[15] the designers of these ovens selected 3-1/2 inches average width on the basis of maximum through-put when allowance is made for charging and pushing time.

Special tests have shown that the volatile-matter content of the finished coke at the midpoint between the oven walls is virtually the same as the volatile matter content of the coke near the oven walls. Further tests showed that if the coke were left in the oven for 2 hours longer than the normal coking time, its properties would not change appreciably and the volatile matter content decreased only about one half of one percent. That is, it decreased from 9.1 to 8.6 percent with the additional 2 hours of carbonization.

With regard to the selection of coals best suited to low-temperature carbonization in these ovens, the most interesting coals in Germany are those with a volatile-matter content of 27 to 38 percent. Besides giving a low-temperature coke of excellent properties and a high tar and light oil yields, these coals are not especially suited to high-temperature coking. That is, unless blended with low-volatile coals, many of the coals in this range do not form coke strong enough for metallurgical use.[16]

Special Tests on Several Coals

Special experiments on pilot-plant scale with different coals showed that when a gas coal of 30 percent volatile-matter content was mixed with 25 percent of a fat coal of 25 percent volatile-matter content, the drum stability was increased from 68 percent on a 1.58-inch screen to 72 percent. The tar yield, based on dry coal, was reduced from 6.4 percent to 6.0 percent. When the gas coal was carbonized by itself, some sponge coke was formed in the plane midway between the heating walls. This sponge coke, however, was not soft enough to lower appreciably the drum test on the coke. Some sponge coke was also formed on the top surfaces of the heating cells under the cover of the ovens. The addition of 25 percent of fat coal eliminated the sponge coke. It was found, also, that the gas coal could be carbonized without admixture if it were tamped into the oven so as to raise the bulk density of the charge.[17]

15/ Mueller, F., Low-Temperature Coking of Bituminous Coal: Oel and Kohle, vol. 12, 1936, pp. 543-549.

16/ See footnote 5.

17/ See footnote 5.

Another experiment was made on coal of 33 percent volatile matter content that was poorly coking. When crushed to a normal fineness of 78 to 80 percent to through 1/8 inch-screen and charged to the ovens, only a very soft coke was formed, which adhered to the carbonization cell walls. When this coal was charged in a screen size of only 62 percent through an eighth and 1.8 percent through 100-mesh screen (bulk density of the wet coal, 56 pounds per cubic foot), a relatively strong coke with a drum stability of over 50 percent was formed. The crushed coke showed a yield of 68 percent on a 2-inch screen and about 15 percent breeze (through a 1/2-inch screen). This experiment indicates that coals ranging widely in their carbonizing properties can be made into a satisfactory low-temperature coke by controlling the bulk density of the charge to the oven.

Some oven-scale tests were made in the Saar on various coals in that district. The low-temperature coke made entirely from Velsen coal (see table 1) has a uniform structure and shows no tendency to form pieces of "half-oven" length. Coke made from mixtures of Velsen coal and more strongly coking coal (Heinitz) has small cell structure near the oven wall and coarser cell structure in the center. Coal from the Saarschacht wine, which is considered noncoking (oxygen content over 10 percent), yielded a powder in the low-temperature coke oven. Coal from the Heinitz mine yielded a weak coke with coarse cell structure. One-half to two-thirds Heinitz coal with one-half to one-third Saarschacht yielded a product similar to Velsen coal. This shows that a strongly caking fluid coal, if mixed with one-third of noncoking coal, yields a satisfactory product.

With regard to the carbonization of briquettes in the Krupp-Lurgi low-temperature carbonization ovens, successful trial runs were made, carbonizing in these ovens briquettes made from noncoking bituminous coal. Specimens of the carbonized briquettes, ovoids about 1 inch long, were dropped on the concrete from a height of 6 feet without breaking. These briquettes were reported to have been made with 5 percent coal-tar pitch for a binder and to have given no trouble by deforming or agglomerating during carbonization. The carbonizing time was only 2-1/2 hours at 600° C. A lot of 5 tons of briquettes made from bituminous coal without binder were also carbonized at the Saar plant, with a coking time of about 2-1/2 hours. No difficulties were encountered in this operation, and briquettes made a firm carbonized product.

Rather interesting experiments were made to determine the composition of the volatile-matter content of the low-temperature coke.[18] The coke was heated in laboratory equipment to 1,000° C. by the Bauer procedure. The resultant yield of distillation products (dry-coke basis) is shown in table 4.

The gas constituting 6.48 percent by weight of the dry coke had the following composition: CO, 7.4 percent; H_2, 77.2 percent; CH_4, 12.5 percent; unsaturates, 0.3 percent, N_2, 2.6 percent, density, 0.223 percent.

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[18] See footnote 5.

TABLE 4. - Distillation products formed when low-temperature coke is heated to 1,000° C.

	Percent by weight
Gas (exclusive of CO_2, H_2S, C_6H_6)	6.48
Carbon dioxide	0.92
Hydrogen sulfide	0.06
Lower boiling hydrocarbons	0.03
Ammonia	0.27
Tar	0.06
Water of constitution (liquor)	1.75
	9.56

From the viewpoint of production of smokeless fuel, it is interesting to note that the tar yield of the original coal is reduced 99 percent by low-temperature carbonization; that is, from 6 percent to 0.06 percent.

Other laboratory experiments were made on a number of coals in a cast-iron retort.[19] The retort carbonized the coal in a sheet about 3 inches thick and contained about 12 pounds of coal.[20] The results of tests in this apparatus on six coals ranging in volatile matter content from 19 to 38 percent are given in table 5.

TABLE 5. - Carbonizing properties of several coals at 600° C.

Coal	A	B	C	D	E	F
Proximate analysis of the coals:						
Water, percent	8.6	8.2	9.6	7.6	9.2	9.4
Ash, percent	10.5	5.6	4.8	6.4	7.7	5.7
Volatile-matter content, percent	19.0	22.8	26.	30.3	33.9	38.0
Yield based upon dry coal:						
Tar, percent	3.2	4.7	6.3	8.6	8.9	10.8
Benzene	0.4	.5	.5	.7	.7	.8
Coke, percent	88.4	86.5	82.8	78.3	77.3	74.2
Liquor, percent	2.1	2.5	2.8	3.0	4.0	5.3
Gas, percent	5.9	5.8	7.6	9.4	9.1	8.9
Percentage of tar boiling below 360° C.	53.1	50.5	60.6	62.1	68.0	63.9
Percentage of tar acid in tar	11.2	12.5	16.0	23.4	30.4	33.0
Volatile matter in coke, percent	6.9	8.0	8.5	8.8	9.8	10.5

19/ Schoeben, K., Laboratory Apparatus for Making Low-Temperature Coke Between Hot Plates: Forshungsberichte Technisho Mitteilungen Krupp, January 1940, pp. 39-44.

20/ Schoeben, K., Analytical Plant Control of Low-Temperature Coke Plant of Krupp-Lurgi Design: Oel und Kohle, vol. 22, 1943, pp. 205-212.

It is interesting to note how the tar yield increases with increasing volatile matter of the coal and also the striking increase in tar-acid yield with increasing volatile matter in the coal.

A series of laboratory experiments on various coals to determine the tar yields showed that by the Fischer-Schrader method tar yields as high as 16 percent (dry basis) can be obtained from some coals. Comparison of tar yields obtained in the commercial-scale low-temperature carbonization plant and the tar yields given by the Fischer retort show that the large-scale plant obtained about 73 percent of the tar shown by the Fischer retort assay.

CONCLUSIONS

The Krupp-Lurgi low-temperature plant can be used for producing a low-temperature coke from a wide variety of coals or from briquettes. There have been many improvements in design since the 4-ton-per-day pilot plant was in operation in 1936. The latest improvements were incorporated in the 24 new ovens built in 1943 in the Ruhr. Further improvements would be made in a new plant. For example designers state that in the future the oven length in a new plant would be 10 to 13 feet and that with these ovens a throughput would be obtained that would compare with the modern high-temperature coke ovens. The method of charging and of discharging the ovens is to be modified. The low-temperature coke is a 50 percent porosity fuel that is a highly reactive, easily ignited, smokeless product containing 8 to 10 percent volatile matter, which should make an ideal domestic fuel. Those in a position to know have stated that a 1,000-ton-per-day plant could be built at approximately the cost of a high-temperature carbonization plant of the same capacity. The heat requirements of low- and high-temperature plants are essentially equal. The data available on other operating costs indicate that under favorable conditions low-temperature coking by a process similar to the Krupp-Lurgi might prove practicable in this country.

However, it should be kept in mind, when considering the economic practicability of the low-temperature carbonization process, that it produces a better domestic fuel than high volatile bituminous coal, and that this better fuel costs more per B.t.u. than the raw material from which it was made. This increase in cost per B.t.u. depends on the size of the plant, the local conditions, and the quality of the coal. All these factors can vary through a considerable range.